APPENDIX C
LANL – HAZARDS TESTING OF INEEL PIT 9 SURROGATES

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Title: HAZARD TESTING OF INEL PIT 9 SURROGATES

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HAZARD TESTING OF INEL PIT 9 SURROGATES

ABSTRACT

Five surrogates, representative of the likely constituents and approximate compositions expected to exist in INEL's disposal Pit 9 were prepared and tested on standard High Explosive Impact and Friction test apparatus to estimate the sensitivity of these compositions to explosive initiation. Thermal behavior of the materials (Modulated-DSC) was also determined.

EXPERIMENTAL

<u>Materials.</u>

The following surrogates were made up in the laboratory, using analytical grade reagents.

A. Surrogate Series 745 Sludge (Mixed nitrate salts):

> Sodium nitrate (6.0 g) and potassium nitrate (3.0 g) were dissolved in the minimum amount of water and the solution was evaporated to a solid on a rotating evaporator. The solid was removed from the flask, placed in an evaporating dish, and dried overnight in a vacuum oven at 80°C.

B. Surrogate Series 745 Sludge containing 13 wt-% Regal R&O 32 oil

> The mixed nitrate salts (2.61 g) and Regal R&O 32 oil (0.39 g) were mixed in a mini ball mill.

C. Surrogate Series 745 Sludge containing 9 wt-% Regal R&O 32 oil

> The mixed nitrate salts (2.73 g) and Regal R&O 32 oil (0.27 g) were mixed in a mini ball mill.

D. Surrogate Series 745 Sludge mixed with graphite

> The mixed nitrate salts (2.13 g) and graphite powder (0.87 g) were mixed in a mini ball mill. This is a CO_2 balanced mixture (71% nitrates/29% graphite).

E. Surrogate Series 745 Sludge mixed with sawdust.

> Sodium nitrate (6.0 g) and potassium nitrate (3.0 g) were dissolved in the minimum amount of water (~13ml) and the solution was poured into a beaker containing sawdust (3.0 g). After soaking for 30 min., the excess solution was decanted and the wet sawdust was dried in vacuum oven at 80°C overnight. The dry weight of the solid was 8.0 g, which translates to a mixture of 62.5% nitrates and 37.5% wood. The CO_2-balanced mixture is 69 % nitrates and 31% cellulose.

F. Standard Materials. HMX (1,3,5,7-tetraaza-1,3,5,7-tetranitrocyclooctane), Holston Defense Corporation, and PETN (pentaerythritol tetranitrate), DuPont Explosives manufacturing, were analyzed according to DOE and Military specifications. All standard materials were from a single lot kept under controlled storage conditions.

Testing Procedure.

Impact Sensitivity

The samples were tested for impact sensitivity using a LANL-modified ERL design (Figure 1.). The apparatus consists of a free-falling 2.5 kg. weight, tooling to hold the explosive sample, and a supporting frame.

An electronic monitoring circuit is used to distinguish between events and failures. The noise that the event creates (>120 db) is picked up by two microphones whose output is fed to a triggering circuit. The threshold of this circuit is adjusted to place one of the standard explosives, usually TNT or HMX, at a fixed point on the drop weight scale.

The sample to be tested is dried and 30–40 mg. loaded into a dimple in the center of a 6.5 cm^2 sheet of 130 N garnet paper (Type 12 configuration). Occasionally a variant, without the garnet paper (Type 12B) is also used. Granular explosives are tested as received; cast explosives such as Comp B, are ground and the sample sieved prior to testing (USS 16, 30, and 50).

A standard series of tests consists of 25 shots performed following the "Bruceton up-and-down:" technique normally employed in sensitivity testing: The results are reported in terms of the height at which an event occurs 50% of the time. The interval between normalized drop heights used at LANL is 0.05 x log (drop height). This interval is added to or subtracted from the $\log_{10}$ of the preceding drop height to determine the $\log_{10}$ of the next drop height. [The logarithmic scale is used on the assumption that the heights at which events occur follow a lognormal distribution.] The maximum height attainable on the impact machine is 320 cm. Because impact sensitivity is affected by humidity, temperature, and minor changes in the apparatus, impact data are always reported along with that of a standard explosive, typically a well-tested lot of HMX.

Friction Sensitivity Testing

Friction sensitivity testing is performed on the BAM (Bundesanstalt für Materialprüfung) test apparatus (Figure 2.) The sample is placed on a roughened 25 x 25 x 5 mm porcelain plate, which is rigidly attached to the sliding carriage of the testing machine. A cylindrical-shaped porcelain pistel, 10 mm in diameter and 15 mm long, with a rough spherical end surface (radius of curvature 10 mm), is placed on top of the sample; the rod is tightly clamped and may be loaded with different weights. The pistel load may be varied from 0.5–36 kg. As with the impact testing, a standard series of 25 tests is performed, with the applied weight varied according to the lognormal procedure described above. A standard explosive—typically PETN—is tested on the same day to ensure comparability under similar conditions of temperature and humidity.

Modulated DSC

A small amount of the mixture (1–3 mg) is weighed in a small aluminum pan and placed on the instrument (TA Instruments, Inc., Model DSC 2920 Modulated DSC™). The sample is heated at a rate of 5 °C/min with a short-period modulation to discriminate between reversible (e.g., melting, phase change) and irreversible (e.g., decomposition) heat flow. Integration of the resulting irreversible heat flow graph gives the heat of decomposition.

RESULTS AND DISCUSSION

The surrogates were made from materials known to exist in the disposal area. While the exact compositions (and likely compositional variations) are unknown, these materials were formulated using a best guess as to the relative amounts of constituents in the pit. To simulate the most powerful explosive composition, an additional formulation was made that approximated a zero oxygen balance (i.e., all the available oxygen from the oxidizer is consumed by the fuel to make CO_2 and H_2O). The optimum composition was determined to be 91 wt-% nitrates and 9 wt-% oil. The impact and friction sensitivity test results are shown in Tables 1 and 2, below.

Table 1. Impact data on Nitrate/organic surrogates.

Sample	H_{50} (cm)	σ Log Units	% RH	Temp (° C)
Nitrates/Wood (62.5/37.5)	>320	N/A	50.3	23.6
Nitrates/Graphite (71/29)	>320	N/A	51.4	23.5
Nitrates/Oil (87/13)	>320	N/A	51.4	23.4
Nitrates/Oil (91/9)	>320	N/A	57.0	23.0
HMX Standard (HOL 41-57)	24.84	0.035	58.7	22.6

Table 2. Friction Sensitivity Data on Mixed Nitrate/organic surrogates.

Sample	50% Load (Kg)	σ Log Units	% RH	Temp (° C)
Nitrates/wood (62.5/37.5)	>36.0*	N/A	54.8	22.9
Nitrates/Graphite (71/29)	>36.0*	N/A	54.2	23.1
Nitrates/Oil (87/13)	>36.0*	N/A	53.9	23.0
Nitrates/Oil (91/9)	>36.0*	N/A	52.9	23.2
PETN Standard	8.4	1.376	53.7	23.2

For comparison, the variability of HMX (Holston Lot 41-57) to impact sensitivity over time is shown in Table 3.

* Tested in the type 12A configuration

Table 3. Variation of Impact Sensitivity of HMX

Test Date	H_{50} (cm)	σ (log units)	%RH	Temp (°C)
12/9/98	26.92	0.043	27.3	18.6
2/26/99	23.76	0.047	23.5	20.4
3/5/99	26.16	0.030	21.0	20.0
3/11/99	25.42	0.063	22.7	20.6
3/26/99	26.16	0.036	30.0	22.0
4/2/99	20.58	0.034	31.0	20.9
4/8/99	24.93	0.047	24.8	19.6
4/20/99	23.31	0.058	23.9	22.2
5/25/99	25.42	0.034	35.1	22.7
6/4/99	24.69	0.085	25.6	21.9
6/18/99	22.87	0.062	45.8	23.0
7/2/99	23.54	0.026	40.0	25.9
7/16/99	27.44	0.080	54.0	21.8
7/22/99	24.84	0.035	58.7	22.6

While some variability in the impact sensitivity is expected, the value should remain fairly constant; for the data in table 3, the mean and standard deviation are: 24.72 ± 1.80 cm. This represents good day-to-day agreement for a semiquantitative test. The variability of PETN friction sensitivity—shown in Table 4— is much higher: 9.4 ± 2.8. The higher variability may be due to the slightly more subjective nature of the event interpretation: a friction-induced event may be deduced from explosions, snaps, crackling, or scorch marks on the ceramic plate.

Table 4. Variation of Friction Sensitivity of PETN

Test Date	50 % Load (kg)	σ (log units)	%RH	Temp (°C)
1/19/99	14.07	2.0561	19.7	19.8
4/2/99	13.85	3.07	12.2	21.1
4/6/99	13.20	3.99	27.8	17.5
4/21/99	9.054	4.421	16.6	22.7
4/26/99	9.707	8.074	26.9	19.3
5/24/99	8.20	2.368	35.5	23.5
6/10/99	6.00	2.718	14.3	23.9
6/18/99	8.62	0.12	47.7	22.7
7/1/99	7.80	3.417	38.0	25.9
7/16/99	7.6	2.9127	54.3	22.7
7/22/99	8.4	1.376	53.7	23.2
8/11/99	6.0	2.6794	57.8	21.4

The fact that none of the mixtures gave any positive events, even at the limitations of the machines, is an indication that if the mixtures are explosive at all, then the stimulus required to initiate them will be extreme. The results cannot, however, be construed to mean *by themselves*, that the mixtures are not explosive at all. For instance triaiminotrinitrobenzene (TATB) is an extremely insensitive explosive that gives no events on the impact or friction sensitivity machines under identical conditions; yet properly initiated, TATB's explosive power is greater than that of

TNT. A more important indicator of explosive nature is the thermal behavior of the mixture. If, under the conditions of rising temperature, the sample undergoes a strong exothermic behavior (e.g., > ~500 cal/g or 2100 J/g), it may be considered to be an explosive. The DSC (differential scanning calorimetry) data for these mixtures are shown in Table 5.

Table 5. Modulated DSC Results for Nitrate salts/organic mixtures.

Sample	Onset of Melting (°C)	Onset of Decomposition (°C)	Heat of Decomposition (J/g)
2/1 $NaNO_3/KNO_3$	217	None	N/A
91/9 Nitrates/oil	217	none	N/A
87/13 Nitrates/oil	216	none	N/A
62.5/37.5 Nitrates/wood	216, 335	306	894.3
71/29 Nitrates/graphite	218	none	N/A

In the case of the mixed nitrates and wood, a reaction occurring during the heating may result in the formation of a small amount of the known explosive nitrocellulose, which could account for the thermal decomposition at 306 °C.

A difficulty with these experiments is that the sample size (~1 mg) is small enough that heterogeneous explosives such as ANFO (ammonium nitrate/fuel oil) do not give consistent results. For these materials, a test utilizing a larger sample, such as the Henkin test (time to explosion), or the Accelerating Rate Calorimeter (ARC) is more useful because the mass of the sample is sufficient to provide an consistent indication of explosive behavior, even though the thermal parameters derived from modulated DSC are inherently more accurate. Nonetheless, the data available are sufficient to suggest that the mixtures tested are not likely to be good explosives, and more importantly, will be difficult to initiate by mechanical (impact, friction) means. Without more detailed thermal testing (e.g., Henkin, ARC), it is difficult to draw conclusions regarding the thermal sensitivity of these mixtures.

CONCLUSIONS

Sensitivity and limited thermal testing of five compositions consisting of mixed potassium and sodium nitrates with various combustible organics (Regal R&O cutting oil, graphite, and sawdust) could not *conclusively* rule out explosive behavior of the mixtures. However, the testing does indicate that these mixtures are extremely insensitive to mechanical initiation, and are without substantial thermal decomposition energy (on a small scale) indicative of an explosive. Larger-scale thermal testing is necessary to definitively rule these mixtures out as explosives.

REFERENCES

"Cheetah 2.0," L.E. Fried, W.M. Howard, P.C. Souers, Lawrence Livermore National Laboratory, 1998. (UCRL-MA-117541, Rev. 5)

LIST OF FIGURES

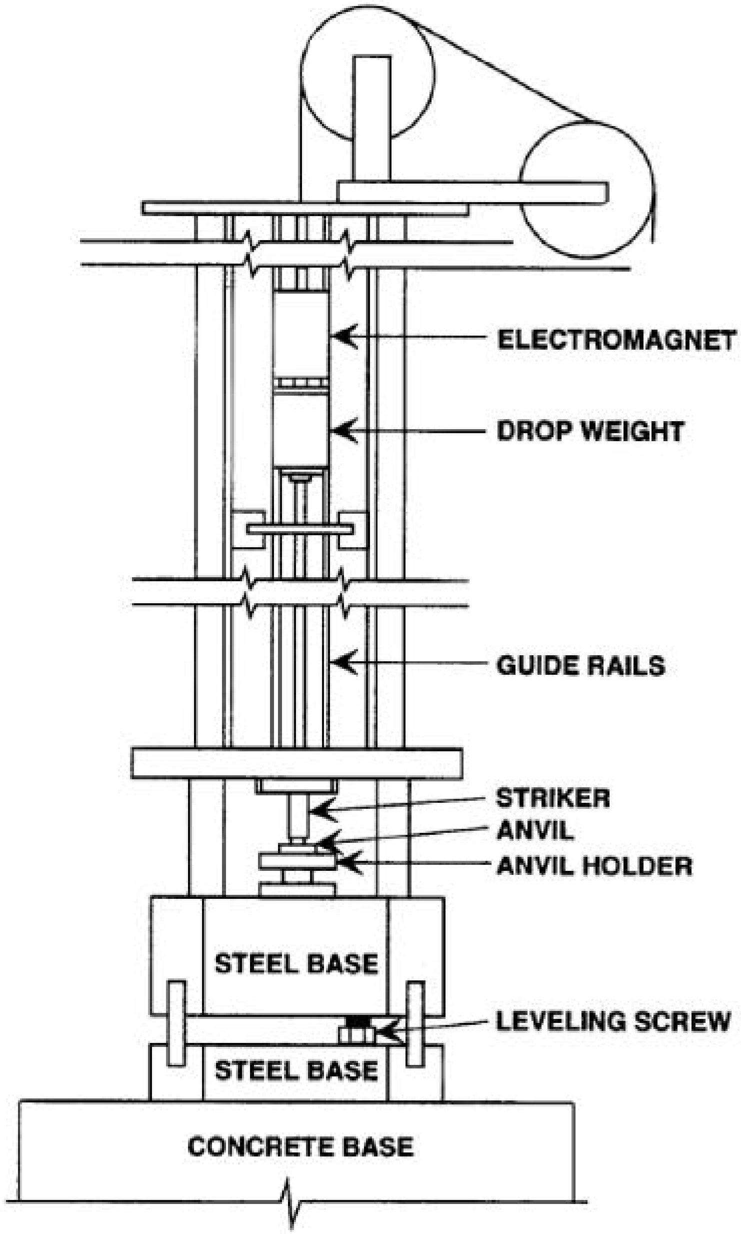

Figure 1. Drop weight impact machine, based on Explosives Research Laboratory model with type 12 tooling

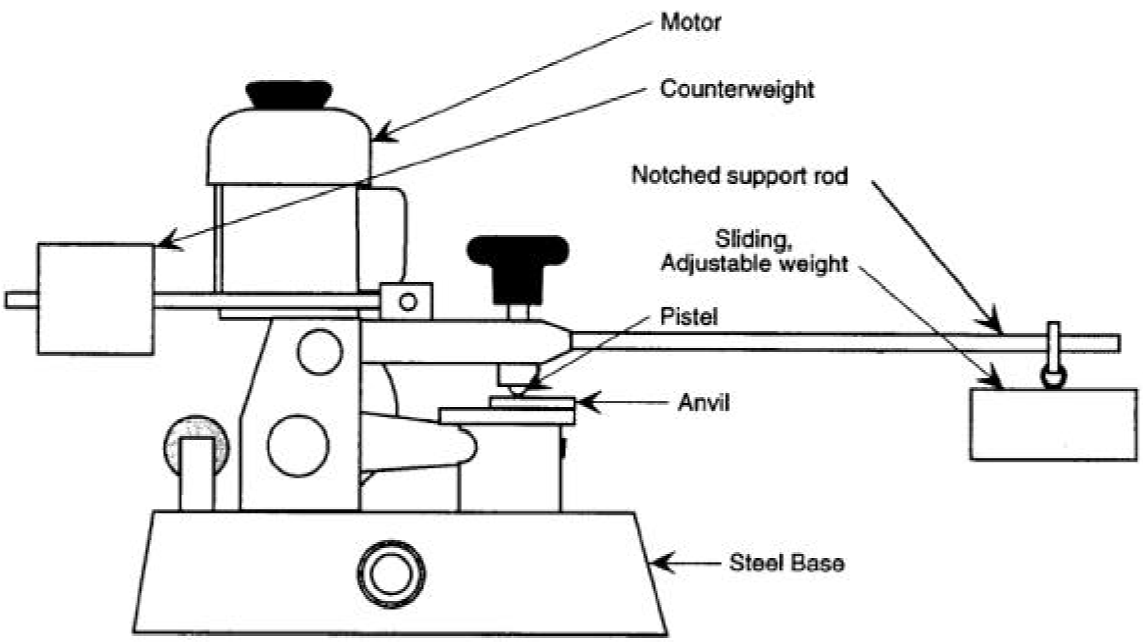

Figure 2. BAM Friction Test Apparatus